



Laboratory Assessment of DoD Coating Adhesion on Ti-6Al-4V

by Tom A. Considine, Chris E. Miller, and Brian E. Placzankis

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14. ABSTRACT Titanium is used in industry to manufacture many consumer products such as softball bats and golf clubs. It is also widely used in aerospace and the defense industries. At issue is that titanium does not maintain a good adhesive bond with organic coating systems. This study evaluates the adhesion of two nonchromate DoD primers, MIL-P-53022 and MIL-PRF-23377 Class N, relative to a direct to metal process using no primer. The evaluation method was based on ASTM D 4541 and used standard hydraulic adhesion testing equipment testing. The effectiveness of each method is then discussed with respect to strength values and failure modes.					
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1. Introduction

With the necessity of titanium and its various alloys on the rise, so accordingly does the need to be able to coat them. Titanium is lighter than but just as strong as steel, twice as strong as aluminum, and extremely resistant to corrosion. It has many current applications, especially in the aerospace industry, and has many more proposed applications on the horizon. One small problem with titanium is that it does not maintain a good adhesive bond with organic coating systems; as such, coating delamination is fairly common. Past studies involving titanium on aircraft have brought this problem to light; therefore, the proposed goal of this report is to find a suitable non-chromate method to help enhance the adhesion of organic coatings on Ti-6Al-4V, or titanium grade 23 (NDCEE, 1997).

2. Experimental Procedure

An 8- × 7- × 1-in block of titanium (Ti-6Al-4V) was pretreated via abrasive blasting, utilizing 60-grit Al₂O₃ blast media, and then divided into three sections for different methods of coating. The top 3-1/4 in was primed according to spec with MIL-P-53022-10 (1992), the middle section received no primer and was masked, and the bottom 2-1/4 in was primed according to spec with MIL-PRF-23377N (2005) (see figure 1). All sections, including the remaining bare section in the middle, were then topcoated with MIL-DTL-64159 (2002) type II at a thickness of approximately 3 mil and allowed to set and cure. After curing, the panel was then cleaned of dust and subjected to the hydraulic adhesion testing equipment (HATE) testing in accordance with ASTM D 4541 (1989). A steel loading fixture (referred to as a dolly) is secured to the surface of the test area using an adhesive under laboratory conditions (see figure 2). In this case, each dolly was set using S-100 Instabond cycnaoacrylate and given a 24-hr period to cure. Only one dolly was adhered to the panel per pull to prevent false data during subsequent pulls due to shock from the initial pulling events. Data from each pull, both adhesive strength and failure mode, were recorded. Pulls that resulted in a failure of the adhesive were ignored.

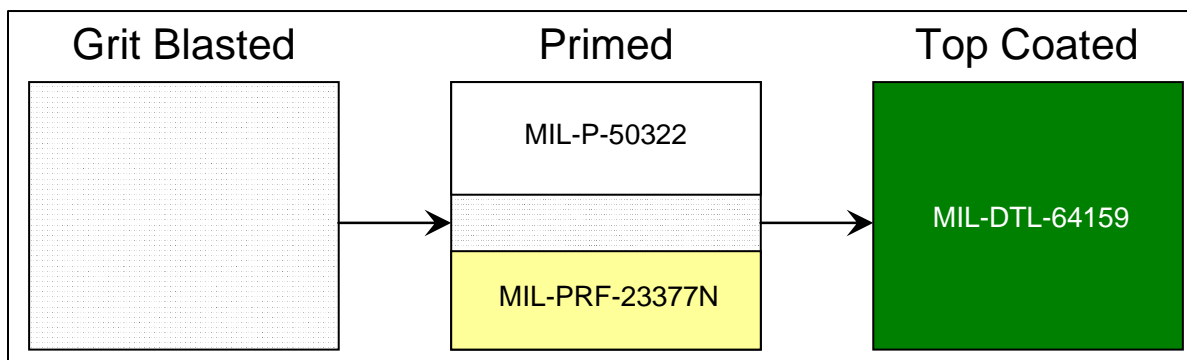


Figure 1. Schematic of surface preparation and coating application steps.

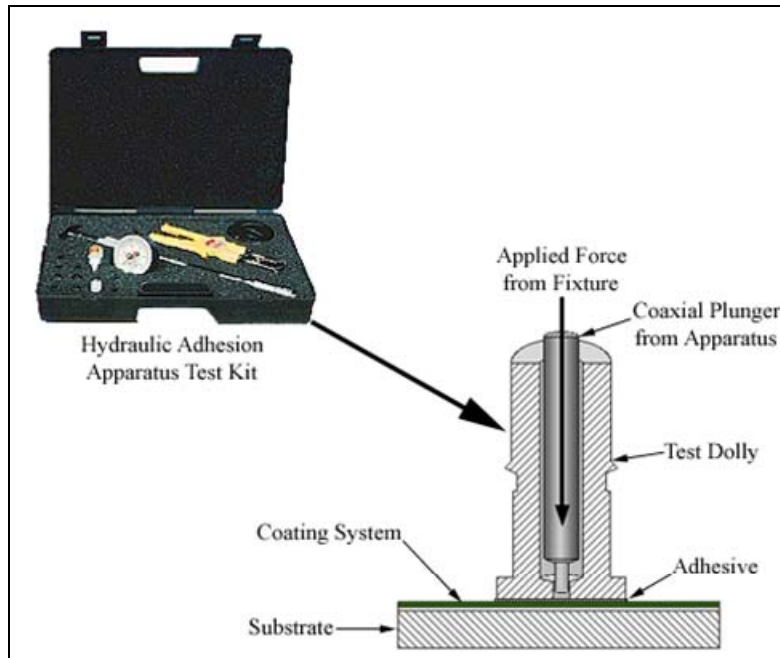


Figure 2. Pull-off hydraulic adhesion test (ASTM D 4541).

3. Results

3.1 MIL-P-53022 Primer

As can clearly be seen from figure 3, the majority of pulls resulted in a cohesive failure of the topcoat. A few pulls resulted however in a slight cohesive failure of the primer around the center where contact was made between the tester and the surface. However, not a single pull-off resulted in any failures at the primer/substrate interface. Adhesive strength was fairly high, maintaining an average pull-off strength of 3545.65 psi (see figure 4). This relatively high average actually called for a slight change in the dolly size for the HATE test. While starting with 3/4-in-diameter dollies, the high strengths were beginning to exceed the capacity of the tester, and forced the use of a larger tester which used dollies of 7/8-in diameter. The switch allowed testing to continue and record maximum value of 4200 psi.

3.2 Grit Blast/No Primer

As can be seen in figure 5, there were a few instances of adhesive failure during the HATE tests. Most of the localization of the failure modes were cohesive within the top coat. The average adhesive strength on this section was 3056.25 psi (see figure 6), about 500 psi lower than the MIL-P-53022 primed section. Here, a maximum adhesion of 3590 psi and a minimum of 1800 psi were achieved.



Figure 3. Post HATE-test MIL-P-53022 section of titanium block.

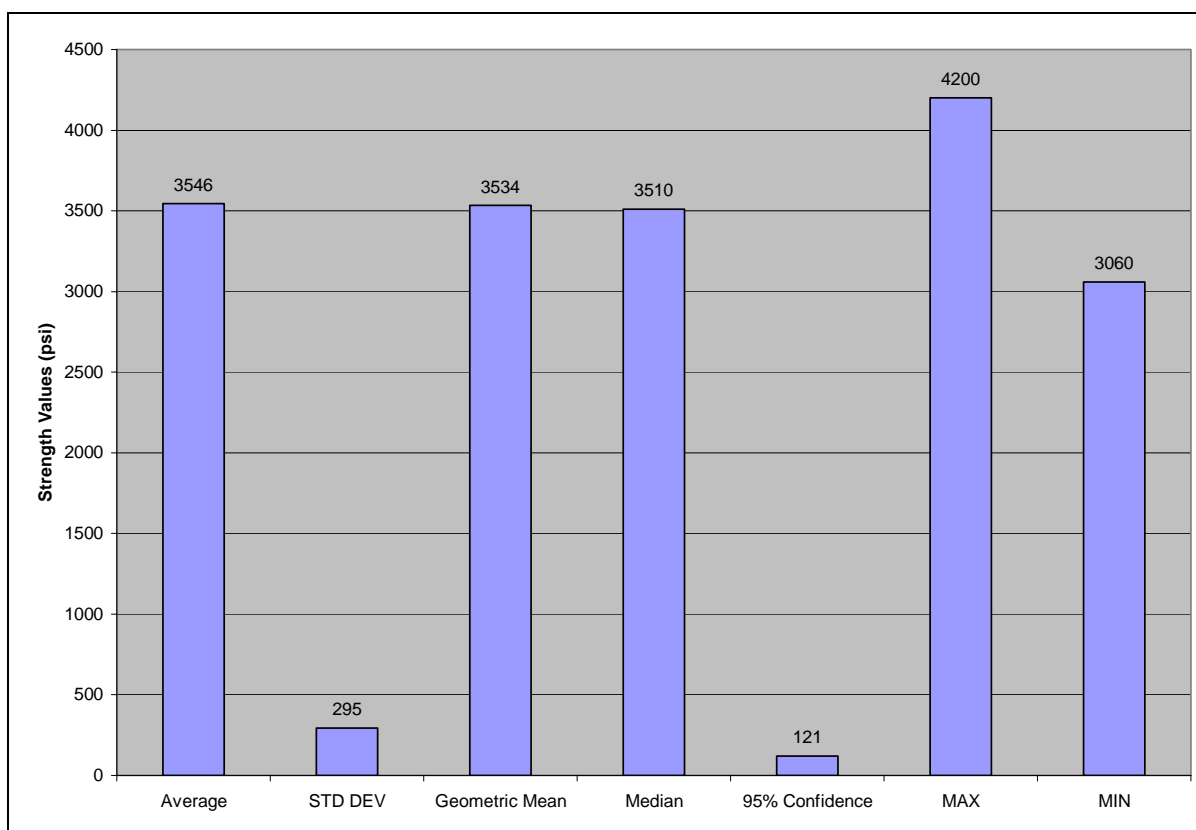


Figure 4. Data obtained from HATE test for MIL-P-53022 primered section.



Figure 5. Post HATE-test grit-blasted/unprimed MIL-DTL-64159 section.

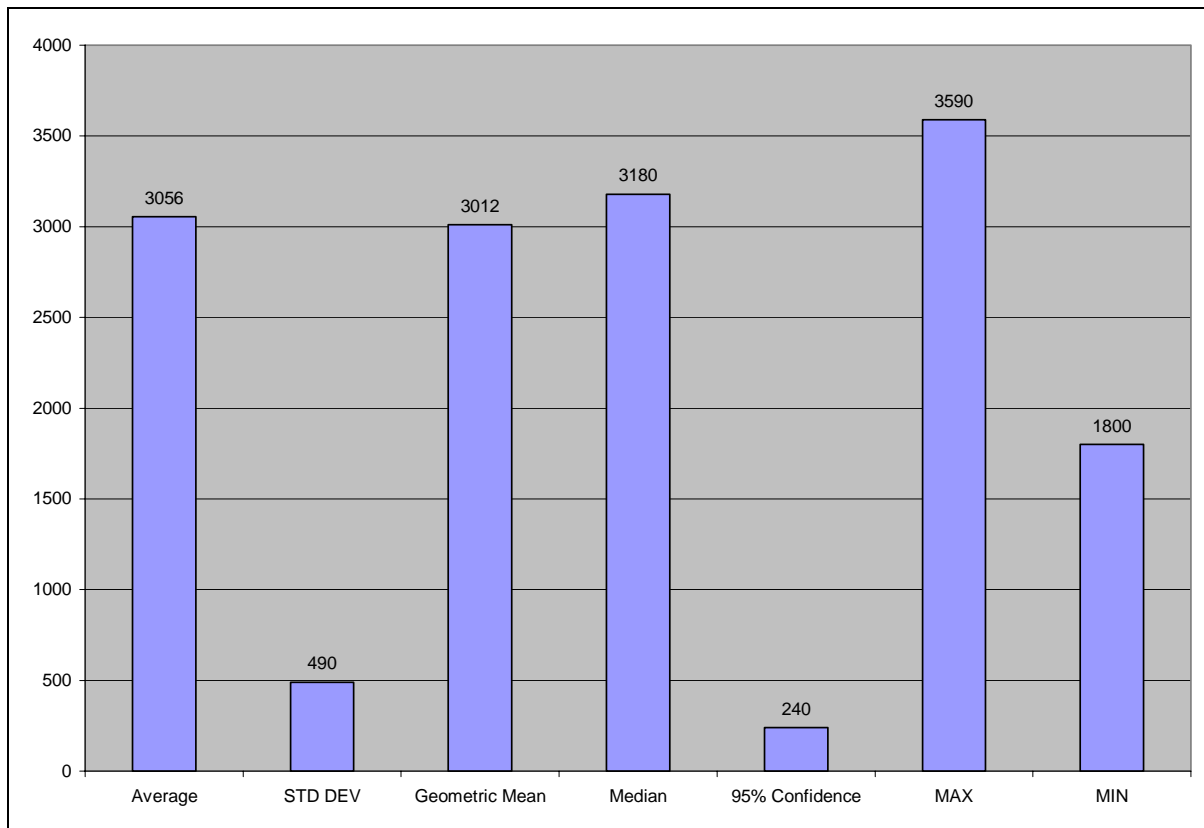


Figure 6. Data obtained from HATE test for abrasive-blasted/unprimed section.

3.3 MIL-P-23377N Primer

A cursory glance at this section reveals that cohesive failures were the majority here (figure 7). Most pulls resulted in a cohesive failure of the primer, while the remainder were cohesive failures in the top coat. Like the MIL-P-53022, not a single pull resulted in adhesive failure at the substrate. An average adhesive strength of 3498 psi was attained, with a maximum at 4200 psi and a minimum of 3100 psi.

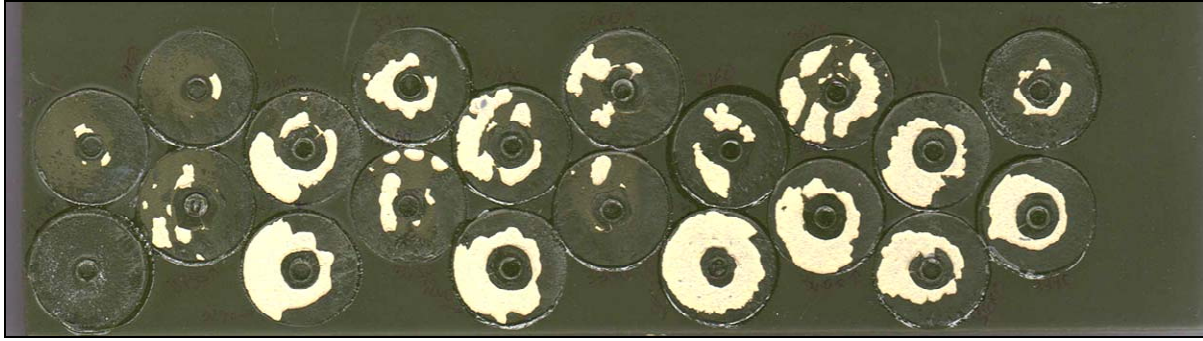


Figure 7. Post HATE-test MIL-PRF-23377N section of titanium block.

4. Discussion

The intent of this study was to find a good, strong non-chromate primer that would properly adhere to titanium armor alloys. It has been found that titanium has an increased susceptibility to delamination; as such, a method for coating titanium armor that addresses and controls this issue was needed. It can be seen from figure 8 that both abrasive blasting followed by priming the titanium substrate with either MIL-P-53022 or MIL-PRF-23377N enhances the substrate's ability to maintain coating adhesion. These findings coincide with a study done by Raytheon Electronics Systems and the Air to Air Joint Systems Program Office, where it was found that using MIL-P-53022 on a grit-blasted or equivalent substrate was the best method in coating titanium. Both primers maintained their adhesion to the substrate under higher strengths, on average, about 500 psi higher than the unprimed section (see table 1). It is interesting to note that each primer/coating performed almost identically when it came to the strength necessary for each pull-off failure. MIL-P-53022 maintained, on average, an advantage of about 50 psi over MIL-PRF-23377N. Although 50 psi is not significant in itself, the way in which each failure occurred needs to be investigated for clarification.

The section of the plate, having only a topcoat applied direct to the abrasive-blasted substrate, was clearly the most susceptible to adhesive failure. In most every pull, at least a small amount of substrate was made visible. Pulls seemed to fail similarly based on their location on the plate. It is possible that these localized failure modes are a result of the uneven nature of abrasive blasting and would explain the results seen in figure 3.

The section primed with MIL-PRF-23377N, as previously stated, performed admirably with respect to strength. However, the majority of the failure modes occurred within the primer layer (see figure 9).

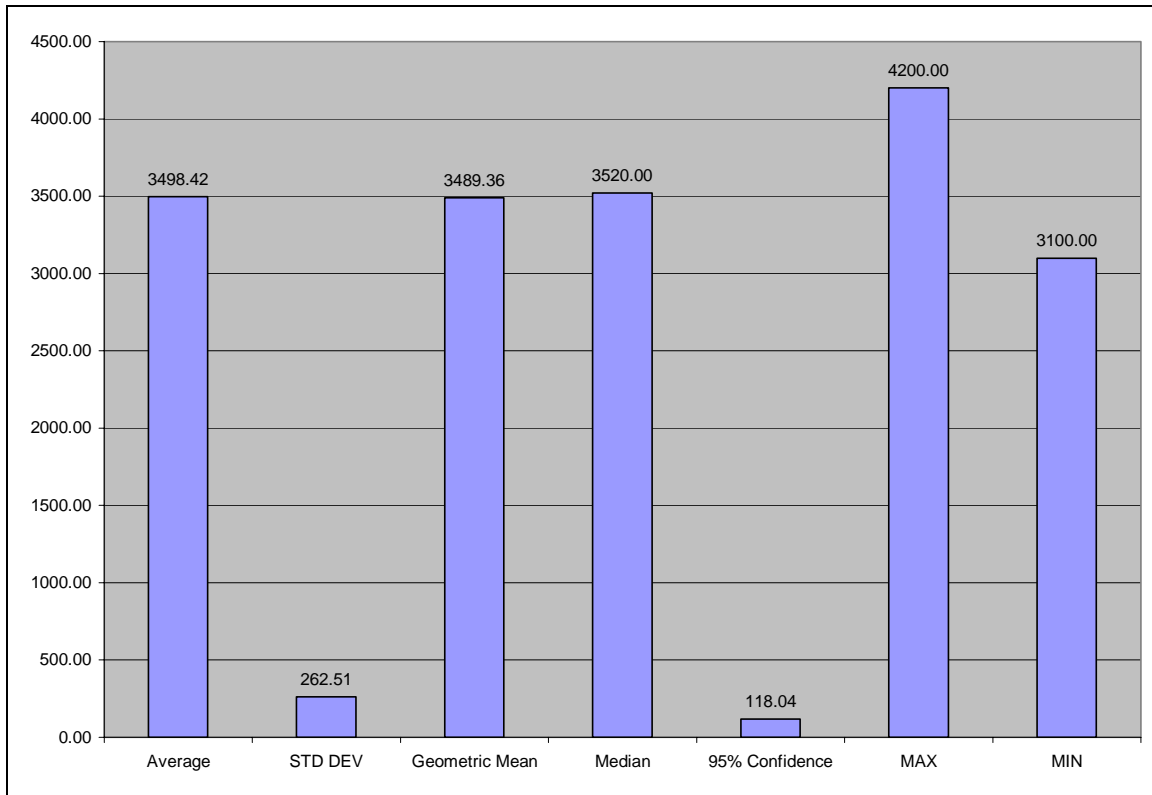


Figure 8. Data obtained from HATE test for MIL-PRF-23377N primered section.

Table 1. Data obtained from HATE testing (values in psi).

	MIL-P-53022	DTM	MIL-PRF-23377N
Average	3545.65	3056.25	3498.42
Std. dev.	295.11	490.45	262.51
Geometric mean	3534.13	3012.04	3489.36
Median	3510.00	3180.00	3520.00
95% confidence	120.61	240.32	118.04
Max.	4200.00	3590.00	4200.00
Min.	3060.00	1800.00	3100.00

It is plainly visible that the grit blast helped to maintain adhesive bond between primer and substrate; however, the primer itself failed cohesively under the stress. Very few of the pulls from the HATE test on this section coated by MIL-PRF-23377N resulted in failures of the top coat. Those that did have cohesive failures on the top coat also had places in the primer that failed cohesively as well.

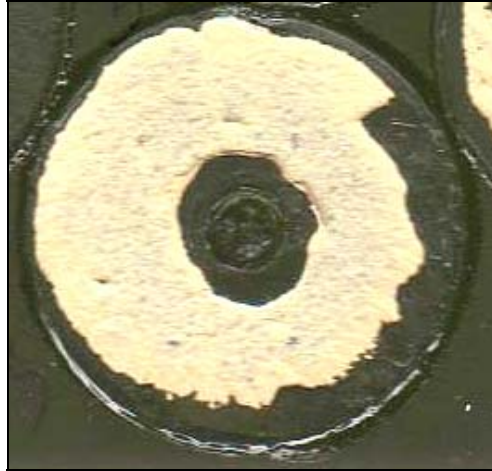


Figure 9. Typical cohesive failure within MIL-PRF-23377N primer.

Finally, the section primed with MIL-P-53022 performed as well as the MIL-PRF-23377N, if not slightly better, with respect to adhesive strength. Each HATE pull within the MIL-P-53022 failed cohesively within the top coat (see figure 10). From this, it can be said that the cohesive nature of MIL-P-53022 involves a stronger bond than that of the MIL-PRF-23377N (see table 2). Both primers, however, did bond well to the grit-blasted substrate, as neither section suffered one single adhesive failure.



Figure 10. Typical cohesive failure of the topcoat applied over MIL-P-53022 primer.

Table 2. Failure mode location breakdown by percent.

	MIL-P-53022	DTM	MIL-PRF-23377N
Cohesive – top coat	100	68.75	15.79
Cohesive – primer	0	NA	84.21
Adhesive – substrate	0	31.25	0

5. Conclusions

1. Pretreating titanium via abrasive blasting worked rather well overall. In the unprimed section, although many of the failures occurred at the substrate-coating interface, the adhesion strength was relatively high.
2. The section primed with MIL-PRF-23377N held even better with higher pull-off values than DTM. The vast majority of the MIL-PRF-23377N included cohesive failures within the primer.
3. The section primed with MIL-P-53022 outperformed the rest, sustaining no adhesive failures and very few cohesive failures within the primer.
4. Since all pulls resulted in cohesive failures in the top coat, it can be said that abrasive blasting followed by priming with MIL-P-53022 is a viable method of coating titanium armor alloys.

6. References

ASTM D 4541. Standard Test Method for Pull-Off Strength of Coated Specimens Subjected to Corrosive Environments. *Annu. Book ASTM Stand.* **1989.**

MIL-P-53022-10. *Primer, Epoxy Coating, Corrosion Inhibiting, Lead and Chromate Free* **1992.**

MIL-PRF-23377N. *Performance Specification, Primer Coatings: Epoxy, High-Solids* **2005.**

MIL-DTL-64159. *Coating, Water Dispersible Aliphatic Polyurethane, Chemical Agent Resistant* **2002.**

National Defense Center for Environmental Excellence (NDCEE). Validation of Alternatives to Chromate-Containing Primer Coatings for Aircraft Exterior Mold Line Skins. Joint Test Protocol MD-P-1-1, December 1997.

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